

Michael O. Leavitt Dianne R. Nielson, Ph.D. Executive Director Don A. Ostler, P.E.

# StateLi-Utah

## DEPARTMENT OF ENVIRONMENTAL QUALITY DIVISION OF WATER QUALITY

288 North 1460 West P.O. Box 144870 Salt Lake City, Utah 84114-4870 (801) 538-6146 Voice (801) 538-6016 Fax (801) 536-4414 T.D.D.

March 20, 1997

Ms. Elaine J. Dorward-King, Ph.D. Director, Environmental Affairs Kennecott Utah Copper 8315 West 3645 South Magna UT 84044-6001

Dear Ms. Dorward-King:

Subject:

Approval of Appendix A and Attached Standard Operating Procedures #1, #2 and #3; Assessment of Acidification Potential; Kennecott Tailings Impoundment; Ground Water Discharge Permit No. UGW350011

The revised version of Appendix A and SOP #1 (attached to your March 7, 1997 letter), along with the previously submitted SOP's #2 and #3 (attached to your letter of January 30, 1997 for the Kennecott Tailings Impoundment) address the deficiencies identified in our earlier correspondence, and discussions between our respective staffs. This plan is approved, and is now incorporated into the Tailings Ground Water Discharge Permit as Appendix A. This approval satisfies the requirements for compliance schedule Item 4, in Part I, Section K of the Tailings Ground Water Quality Discharge Permit.

The approved plan requires an annual report summarizing all of the previous year's sampling results to be submitted on March 31, 1997. Given the date of this approval, the due date for the 1996 annual report will be extended to April 30, 1997. This will allow Kennecott to complete all analysis and compile the data.

Thank you for your cooperation in this effort.

Sincerely,

Utah Water Quality Board

Don A. Ostler, P.E. **Executive Secretary** 

DAO:JW:wlm

CC:

Terry Sadler, Salt Lake County Health Dept.

Wayne Hedberg, DOGM

Gene Farmer

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Keith W. Welch Chairman

Lynn F. Pett

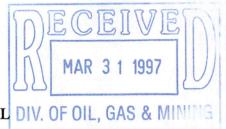
R. Rex Ausburn, P.E. David S. Bowles, Ph.D., P.E.

Nan Bunker Leonard Ferguson Dianne R. Nielson, Ph.D. Joe C. Nielson K.C. Shaw, P.E. J. Ann Wechsler

> Don A. Ostler, P.E. Executive Secretary







# ASSESSMENT OF ACIDIFICATION POTENTIAL DIV. OF OIL, GAS & MINI

(Revised March 7, 1997)

## 1.0 MONITORING OBJECTIVES

The objectives of this monitoring plan are as follows:

- 1. To characterize any potential water quality impacts resulting from future acidification of tailings material.
- Accurately quantify the acidification that will occur on the impoundment to allow DOGM to assess if the reclamation proposed for the impoundment will prevent or minimize wind and water erosion through the establishment of a diverse and self-sustaining vegetative community.
- 3. Present an adequate characterization of acidification potential for the different units of both the existing and expansion portion of the Tailings Impoundment.

## 2.0 MONITORING PLAN

## 2.1 Acidification Monitoring

The primary objective of this portion of the monitoring program is to determine the acidification potential of both the existing and expansion portions of the tailings impoundment, using acid/base accounting (ABA) and kinetic testing.

## 2.1.1 Sampling Locations

The new embankment will be constructed of underflow material from two cyclone stations (designated East and West Cyclones). The West Cyclone Station will independently provide underflow material for construction of the North embankment up until fourth quarter 1998, when the East Cyclone Station becomes operational. During the initial construction phase, overflow material from the West will be discharged to the existing impoundment.

Whole tailings will continue to be discharged to the existing impoundment during construction of the North Impoundment, through completion of reclamation activities for the existing impoundment (i.e. year 2004).

Sampling locations include the following sites, to be sampled at the frequencies indicated in section 2.1.2.

- I. Interior of the Existing Impoundment
  - a. Copperton Concentrator Tailings
  - b. Magna Concentrator Tailings
  - c. Slag Tailings
  - d. Power Plant Ash
  - e. Hydrometalurgical Plant Flow
- II. Embankment of the North Expansion (cycloned tailings underflow)
- III. Interior of North Expansion
- IV. Embankment of Existing Impoundment

## 2.1.2 Sampling and Analysis for ABA Values

Samples will routinely be collected from a depth interval of 0 to 12 inches for tailings that are in place. However additional samples may be taken from other depths for evaluation of areas of incipient acidification.

SOP #3 describes the standard protocol for sampling, preservation, chain of custody and archiving of samples. All samples will be archived for at least two years. The locations of the samples from the existing impoundment will be marked in the field with a stake and will be indicated on a reference map. The samples from the embankment of the existing impoundment will be collected in September, 1996.

## 2.1.2.1 Interior of the Existing Impoundment

- A. Copperton Tailings A monthly grab sample will be collected from this tailings stream for a period of two years for ABA characteristics. After a period of two years, sampling frequency will be reduced to quarterly grab samples if the sample ABA mean identifies the population mean plus or minus 10 tons per 1000 tons at a 95% confidence interval.
- B. Magna Concentrator Tailings A monthly grab sample will be collected from this tailings stream for a period of one year for ABA characteristics. After one year, sampling frequency will be reduced to quarterly grab samples if the sample ABA mean identifies the population mean plus or minus 10 tons per 1000 tons at a 95% confidence interval.
- C. <u>Slag Tailings</u> A grab sample will be collected from this tailings stream once every six months for ABA analysis. The sampling frequency will be reduced after two years to one sample per year if the observed ABA values are greater than +10 tons per 1000 tons or the Acidification Potential values are less than 1 ton per 1000 tons.

- D. Power Plant Ash This material is highly alkaline, and not expected to have much if any potential for generating acid. To verify the ABA characteristics, one sample will be collected from this tailings stream every six months until three samples have been collected. Sampling will be terminated after 3 samples if the observed ABA values are greater than +10 tons/kton or the Acidification Potential values are less than 1 ton per 1000 tons. If the sampling continues beyond the initial 3 samples, one additional grab sample will be collected each year until sufficient samples have been collected to characterize variability.
- E. Hydrometalurgical Plant Effluent This tailings stream consists largely of gypsum at an elevated pH. To verify the ABA characteristics, one sample will be collected every six months. After three samples have been obtained, sampling frequency will be reduced to one sample per year if the observed ABA values are greater than +10 tons per 1000 tons or the Acidification Potential values are less than 1 ton per 1000 tons.
- F. Interior of Existing Impoundment Twelve additional samples will be collected from the top surface of the central area of the existing impoundment after the final lift has been placed. The total of 12 is based on one or two samples from each of the reclaim areas that will be constructed post-closure. The samples will be distributed between the margins and the central area along the reclaim dikes as they are constructed between 1996 and 2004. For the larger areas, one sample will be taken near the margins and one near the center to evaluate possible horizontal variability. As samples are collected and analyzed KUC will analyze the data statistically to determine if the sample ABA mean identifies the population mean plus or minus 10 tons per 1000 tons at a 95% confidence interval. If the ABA samples do not characterize the population mean within the specified limits, KUC shall provide for approval by DOGM, either plans for additional sampling or justification why further sampling is not necessary.

## 2.1.2.2. Underflow Material in New North Embankment

Grab samples of underflow tailings will be collected monthly. The samples will be collected as underflow is discharged from the cyclone. After the first year, underflow ABA values will be evaluated jointly with results from kinetic testing for the ABA zone of uncertainty, statistical mean, standard deviation, and variance. Using standard confidence interval tests, KUC will evaluate the data for variability and trends in the acid potential values of the underflow tailings materials. Following receipt of the annual report including data and analysis, a reduced sampling frequency may be proposed by KUC if the sample ABA mean identifies the population mean plus or minus 10 tons per 1000 tons at a 95% confidence interval.

Construction of the new embankment will be performed using two cyclone stations (East and West cyclone stations); because the West cyclone becomes operational approximately 1 ½ years prior to the East, samples will solely be collected from the West cyclone during this time period and then alternate between the East and West cyclone stations.

## 2.1.2.3 Overflow Material in the New North Impoundment

To characterize the tailings that will be discharged as overflow to the interior of the North Expansion Impoundment, one grab sample of overflow tailings will be collected quarterly from each cyclone station (two samples per quarter). After the first year, KUC will use standard confidence interval statistical tests to evaluate ABA data for all tailings overflow samples to date, including variability and trend analysis. Following receipt of the annual report including data and analysis, a reduced sampling frequency may be proposed by KUC if the sample ABA mean identifies the population mean plus or minus 10 tons per 1000 tons at a 95% confidence interval.

## 2.1.2.4 Embankment of the Existing Impoundment

## A. Upper Section of the Existing Embankment

An additional 20 samples of tailings will be collected and tested for ABA values from a variety of locations on the upper section of the embankment. In addition, five samples will be analyzed for quality assurance; these will include two reference tailings samples, plus three replicates from the 20 new samples.

Six of the additional samples will be taken from a single, small sampling location, for the purpose of better estimating the inherent variability that exists in the tailings within a small spatial area, including the variability in analytical laboratory procedures; the six samples will be taken from an area not greater than about three to five feet in diameter.

The remaining 14 samples will be selected from a variety of locations in the upper portion of the existing embankment. The sampling traverses used in the previous acidification study will be avoided in order to increase the variability of the samples. The purpose of additional sampling will be to further characterize the possible effects of stratification and lateral variation on the ABA values, as well as to test areas that may show incipient acidification on the basis of color or paste pH.

## B. Middle Section of the Existing Embankment

Twelve additional samples will be taken from the middle portion of the existing embankment. Samples will be taken from sites evenly spaced around the embankment but avoiding the transects previously sampled in 1995.

#### C. Lower Section of the Existing Embankment

Ten additional samples will be collected from the lower section of the existing embankment for ABA analysis. These samples will be taken from sites evenly spaced around the embankment but avoiding transects previously sampled in 1995.

Statistical analysis indicates that these additional samples may be required to adequately characterize the lower portion of the existing embankment. The variation in the results to date is effected by carbonate bearing alluvial fill that was mixed into the tailings in the lower section of the embankment; this has resulted in some samples with high ABA values.

## 2.1.3 Testing Methods and Parameters

Samples of tailings solids will be analyzed using methods described in detail in the attached Standard Operating Procedures listed in section 4.0 (SOP's 1, 2, and 3,)

## 2.1.3.1 Static Testing

Samples of tailings solids will be analyzed for acid/base accounting using the protocol for ABA potential in SOP # 1.

## 2.1.3.2 Kinetic Testing

Additional kinetic testing of samples will be conducted in accord with SOP #2. The objective of the kinetic testing will be to identify the range of ABA values below which tailings materials will acidify as a result of exposure to surface weathering. Samples selected for kinetic testing will be replicates or duplicates of the same samples for which the ABA values were determined.

Six samples will be selected for kinetic testing from the samples collected for ABA analysis each year for the first four years. No more than four kinetic tests will be conducted at any one time. Samples will be selected for kinetic testing based on ABA results. Initially, samples with ABA values between +10 and -10 tons/ktons will be utilized. Subsequent kinetic tests will be performed on samples so as to establish what ABA values will become acid producing.

The length of time for kinetic testing of individual samples will be based on the following criteria:

- 1. If the pH of the humidity cell leachate remains at or below 3.5 for 2 weeks, then the test can, at the discretion of Kennecott, be terminated.
- 2. All other tests will be conducted for 20 weeks unless the following conditions are met:

- A. If the pH of the humidity cell leachate drops by 2 or more units between weeks 11 and 20, the test will be extended until the pH stabilizes (defined a changing less than 0.1 pH unit per week) or drops below 3.5. If the pH drops below 3.5 for 2 weeks, the test can be terminated at the discretion of Kennecott.
- B. If the average rate of release of sulfate in leachate from the humidity cell between weeks 16 and 20 is greater than 50 mg/kg of solid sample, and the corresponding calculated rate of acid released by oxidation of pyrite would be enough to deplete the neutralization potential within 52 weeks, the test shall be extended until acidic conditions develop, or until 52 weeks have passed since initiation of the kinetic testing.

## 2.1.4 Quality Assurance

Replicate Samples: Split replicate samples will be included to evaluate the precision of the analyses. At least one replicate sample will be analyzed for every 20 samples (5%). Results will be acceptable if the Relative Percent differences (RPD) of the ABA values differ by less than 35%.

Reference Sample: A large quantity of Kennecott underflow tailings sample has been collected, dried, homogenized, and archived as a reference material. The reference sample is a composite sample of tailings underflow material that has been thoroughly mixed and split into individual samples. (This is the same material as was used in the previous evaluation of the acidification potential of the tailings). Samples of the reference tailings material will be submitted to the analytical laboratory, together with the unknown samples, to determine the precision and consistency of the laboratory analyses. One reference sample will be submitted per 20 unknown samples. The results will be compared to those obtained for the same reference material in the previous evaluation of potential acidification (Shepherd Miller, Inc. And Schafer and Associates, 1995). If the RPD is within  $\pm$  1.5 standard deviations about the mean relative to past results for the same reference materials, the results will be accepted.

## 2.1.5 Inspection of North Embankment

Annual inspection of the North Embankment will be conducted to identify potential "hot-spots." If acidification appears to be developing based upon changes in color or lack of vegetation, the site will be marked on a map and a sample collected for soil pH. In addition, seeps will be measured for pH annually. If the pH of the seep is below 6.0, a follow-up investigation will be conducted to identify the cause of the low pH.

## 2.2 Operational Monitoring

The operational monitoring will be conducted as outlined in Kennecott's Operational Monitoring Plan (see Appendix B).

#### 3.0 REPORTING

Commencing in 1997, an annual report will be submitted by March 31 of each year for monitoring described in this plan and conducted the previous year. This report will include copies of all laboratory analytical results for all samples analyzed during the prior year, statistical analysis, along with quality assurance/quality control results.

The report will present the results of the ABA and kinetic testing. The following items will specifically be included:

- A plot of pH, conductivity, net alkalinity, and cumulative sulfate concentration will be provided for each kinetic test.
- The data and calculations used to justify the termination of kinetic tests will be provided.
- A table will be provided that shows the effect of using the correction factor to adjust for the fraction of the sample lost during the acid leaching steps of the ABA analyses.

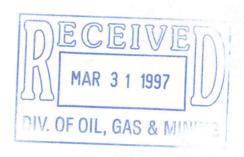
The report will also contain a list of samples proposed for the subsequent years kinetic testing program.

## 4.0 STANDARD OPERATING PROCEDURES (SOP'S)

The following SOP's are attached to this plan:

- 1. Standard Operating Procedure #1 The Complete Modified Sobek Acid Base Accounting
- 2. Standard Operating Procedure #2 Kinetic Testing by the Humidity Cell Procedure
- 3. Standard Operating Procedure #3 Sample Collection, Preservation, Chain of Custody, Archiving, and Quality Assurance

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## STANDARD OPERATING PROCEDURE #1

## The Complete Modified Sobek Acid Base Accounting

Ground Water Discharge Permit No. UGW350011

Appendix A

Revision 4 - March 1997

Prepared by: Kennecott Utah Copper

Kennecott Environmental and			
Industri	al Hygiei	ne Laboi	ratory
9600 W	2100 S	Magna.	UT 84044

SOP NO. KEL-5010.04 **REVISION NO. 4** 

**DATE 03-07-97 PAGE 1 OF 16** 

## STANDARD OPERATING PROCEDURE

THE COMPLETE **MODIFIED SOBEK ACID BASE ACCOUNTING** 

APPROVAL: \_

ality Assurance Manager

Laboratory Director

Date

3/7/97 Date

DATE 03-07-97 PAGE 2 OF 16

#### I. TITLE

## THE COMPLETE MODIFIED SOBEK - ACID BASEACCOUNTING

#### II. SUMMARY

The purpose of this procedure is to measure the acid generating and neutralization potential of waste rock and tailings. This procedure is based on an original procedure developed by A. A. Sobek and others (1978 - EPA Method 600/2-78-054). Several modifications were made to this original method as recommended by Lawrence and others (1989) and White and Others (1996). Other minor modifications were also made to facilitate equipment present at the Kennecott Laboratory.

The acid generating potential results from the presence of metallic sulfide minerals. The sulfides contained in these minerals may be converted to sulfuric acid by oxidation of the sulfur. If sufficient neutralizing capacity is not present, the material may become acidic. This method measures the total concentration of sulfur, differentiates the forms of sulfur present, and measures the neutralization potential. The total sulfur present is determined analytically using a LECO Furnace. The forms of sulfur present are determined by leaching the samples with water, hydrochloric acid, and nitric acid and then analyzing the leached samples for sulfur using the LECO Furnace.

This method is based on the assumption that the forms of sulfur present in the sample can be differentiated as follows:

- Sulfate minerals, primarily gypsum, are soluble in a hot water leach.
- Sulfate and non-acid producing forms of sulfur are removed by a HCl leach.
- Sulfate, non-acid producing forms, and sulfide forms of sulfur, such as pyrite are removed during the HNO<sub>3</sub> acid leach.
- Forms of sulfur remaining after the HNO<sub>3</sub> leach (residual sulfur) will be primarily non-acid forming sulfur compounds and organic sulfur.

Then, by subtraction, the various forms of sulfur can be evaluated. The portion of the sample comprised of sulfitic forms of sulfur (pyritic sulfur) is of primary interest since it is this form that produces acid. The concentration of pyritic sulfur is determined by subtracting the residual sulfur and HCl extractable forms of sulfur from the concentration of total sulfur present in the sample. The hot water leach is optional since sulfate forms are extracted by both the hot water and HCl leach.

The Neutralization Potential (NP) is measured by over-acidifying the sample and then measuring the excess acid and determining the amount of acid consumed by the sample. The net neutralizing capacity of the sample is reported as the Acid Base Account (ABA) of the sample, which is the difference between the NP and the Acid Potential (AP) as determined from the concentration of pyritic sulfur. Total sulfur and the forms of sulfur are reported as percent sulfur, while the NP, AP and the ABA are reported as tons CaCO<sub>4</sub>/1000 tons.

### III. PROCEDURES

Each sample for ABA analysis will follow all of the following procedures.

## A. Sample Preparation

- 1. Apparatus:
  - a) Drying Oven
  - b) Mesh screen
    - (1) All samples will be oven dried at 50°C¹ and homogenized by the laboratory prior to the start of any analytical procedures. All ABA analyses will be run on a portion of sample ground to less than or equal to 200 mesh. Approximately 5 grams of material will be required.

#### B. Total Sulfur

- 1. Reagents and Apparatus:
  - a) Leco Sulfur Analyzer
  - b) Balance with 0.001g readability

Recommeded in ASTM D 5744 9.4

c) Leco Crucible

#### 2. Procedure

- a) Weigh suitable sample aliquot of (0.1-1.0g) into a Leco crucible.
- b) Analyze the sample on the Leco Sulfur Analyzer following the manufactures specifications for sulfur.

## 3. Reporting

a) The result from this procedure is the Percent Total Sulfur.

## C. <u>H20</u> Extractable Sulfur (If requested)

- 1. Reagents and Apparatus:
  - a) Filter rack and glass Funnels.
  - b) S&S 589 white ribbon filter paper or equivalent.
  - c) ASTM Type II Reagent water.
  - d) ml beaker to boil water.
  - e) Microwave to boil the container of water.
  - f) Balance with 0.001g readability.

## 2. Procedure:

- a) Weigh a 1-2g sample.
- b) Fold white ribbon filter paper and place inside the glass funnels.
- c) Transfer sample to the filter paper.

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9600 W. 2100 S., Magna, UT 84044		

- d) Slowly add a total of 100 ml in two or more increments of the boiling water to the sample. Be careful not to lose any sample by run over, splashing or breaking through the filter paper.
- e) Allow the sample to air dry overnight.
- f) The soil sample is analyzed on the Leco Sulfur Analyzer following the manufactures specifications for sulfur.

## 3. Reporting:

a) H2O Extractable Sulfur is reported using Total Sulfur - H2O Extractable in percent

## D. HCl Extractable Sulfur

- 1. Reagents and Apparatus:
  - a) Filter rack and glass Funnels.
  - b) S&S 589 white ribbon filter paper or equivalent.
  - c) Reagent grade HCl/ASTM Type II Reagent water.
  - d) Balance with 0.001g readability.
  - e) AgNO3 10g into 90 ml of ASTM Type II water bring to 100 ml volume. Keep reagent in an amber bottle.

### 2. Procedure:

- a) Weigh a 1-2g sample.
- b) Fold white ribbon filter paper and place inside the glass funnels.
- c) Transfer sample to the filter paper.

- d) Slowly add a total of 100 ml in two or more increments of the 2:3 HCl reagent to the sample. Be careful not to lose any sample by run over, splashing or breaking through the filter paper.
- e) Rinse sample with deionized H2O until all traces of Cl ions are removed. Verify the Cl ion by testing filtrate with 3 drops of AgNO3 to see if a precipitate forms. If a precipitate forms continue washing the sample with deionized water. If no precipitate forms continue with the procedure.
- f) Allow the sample to air dry overnight.
- g) Remove the soil from the filter paper and place in a tarred solo plastic cup. Record the weight of the sample.
- h) The soil sample is analyzed on the Leco Sulfur Analyzer following the manufactures specifications for sulfur.
- i) Weight Correction:
  - (1) This procedure is done to correct for weight loss due to dissolution during leaching the sample, since the Leco sulfur apparatus reports percent sulfur on the actual weight of the leached sample used for thedetermination
  - (2) Correction for the amount of sample dissolved is accomplished as in the following example:

% Sulfur Corrected = % Sulfur Uncorrected times (1-(Initial weight-final weight)/initial weight).

#### Example:

Sample Weight before washing = 2.3211 Sample Weight after washing = 1.6922 Difference = 0.6289

Kennecott Environmental and			
Industrial Hygiene Laboratory			
9600 W 2100 S., Magna, UT 8404			

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0.6289/2.3211 = 0.27

Sulfur = 2.78

Then 2.78(1-0.27) = 2.03

## 3. Reporting:

a) Report (Total Sulfur-Weight corrected Sulfur in the extracted sample) as HCl Extractable %.

## E. HNO3 Extractable Sulfur

- 1. Reagents and Apparatus:
  - a) Balance with 0.001g readability
  - b) Hot plate.
  - c) Beakers.
  - d) Reagent grade HNO3/H2O
  - e) Type II Reagent Water

### 2. Procedure:

- a) Weigh a soil sample of 1-2g of less than or equal to 200 mesh into a 150 ml beaker.
- b) Add 50 ml of 1:7 HNO3/H2O.
- c) Place sample on hot plate. Heat to near boiling cover and Continue to digest for six hours. (Do not allow sample to become dry.)
- d) Let samples cool.

- e) Filter the sample and rinse thoroughly to remove all traces of NO3 (approximately 200 ml of H2O).
- f) Allow the samples to air dry.
- g) Remove the sample from the filter paper and place in a tarred solo plastic cup. Record the weight of the sample.
- h) Carefully transfer the sample to a Leco crucible.
- i) Analyze the sample on the Leco Automatic Sulfur Analyzer following the manufactures specification for sulfur.
- j) Weight Correction:
  - (1) This procedure is done to correct for weight loss due to dissolution during leaching the sample, since the Leco sulfur apparatus reports percent sulfur on the actual weight of the leached sampleused for the determination.
  - (2) Correction for the amount of sample dissolved is accomplished as in the following example:

% Sulfur Corrected = % Sulfur Uncorrected times (1-(Initial weight-final weight)/initial weight).

## Example:

Sample Weight before washing = 2.3211 Sample Weight after washing = 1.6922 Difference = 0.6289

0.27

Sulfur = 2.78

Then 2.78(1-0.27) = 2.03

## 3. Reporting:

a) Report as (Total Sulfur-Weight corrected Sulfur in the leached sample) as % HNO3 Extractable.

## F. Residual Sulfur:

1. Residual Sulfur is calculated as follows:

Residual Sulfur = Total Sulfur - (HCl Extractable + HNO3 Extractable Sulfur)

## G. Calculation of Acid Potential as Tons CaCO3/1000 tons soil.

1. Calculation:

Acid Potential = HNO3 Extractable Sulfur - HCl Extractable Sulfur \* 31.25

• is a factor which yields the Acid Potential in terms of tons CaCO3/1000 tons of material.

## H. Neutralization Potential - Lime as CaCO3

### 1. Summary

The neutralization potential is to determine the neutralizing bases, including the carbonates, present in a given sample. A known volume of HCl is added to the sample. The sample is heated to ensure that the reaction goes to completion. The CaCO3 equivalent of the sample is obtained by determining the amount of unconsummed acid by titration with standardized sodium hydroxide.

## 2. Apparatus:

- a) 100 ml beakers
- b) Mettler DL 70 Autotitrator equipped with ST20 Autosampler
- c) pH meter

## 3. Reagents:

- a) 1:3 HCl/H2O
- b) 0.1 N HCl certified.
- c) 0.5 N HCl certified.
- d) 0.25 N NaOH certified.
- e) Potassium Acid Phthalate primary standard grade.

### 4. Standardizing Reagents:

- a) Standardize the NaOH solution by weighing 1.00g Potassium acid phthalate into three disposable beakers. Write down the exact weight and enter into the titrator program when requested. Titrate with certified 0.25 NaOH to the break point using the DL70 autotitrator and DL70 routine CAL1. The routine will add sufficient DI water, titrate to the dynamic<sup>2</sup> end point, and calculate the NaOH normality automatically.
  - (1) Verify the standardization of the Hydrochloric Acid solution by placing three 10 ml aliquots of the HCl solution

<sup>&</sup>lt;sup>2</sup> The dynamic endpoint is that part of the titration curve where the slope is at a maximum. It has been found that when particulates are present in the sample, this means of end point determination yeilds results that are much more reproducible and accurate than a preset endpoint. Unfortunately this requires the use of an autotitrator with this capability.

into a plastic disposable beaker. Titrate to the end point using routine CAL 3. The method calculates the HCL normality automatically.

## 5. Procedure:

- a) Add 10 ml of 0.50 N HCL slowly to the sample.
- b) Add 75 ml of deionized water and reflux (Term used to describe the liquid condensing from the rising vapor and allowed to flow droplet by droplet back to the liquid it came from. Typically performed using a watch glass or a conical flat bottom flask.) for 1 minute to drive off CO2. The reaction is complete when no gas evolution is visible and particles settle evenly over the bottom of the flask. Let cool.
- c) Calibrate the pH meter according to the pH manual.
- d) Set cooled sample on the stir plate with the stir bar in the sample. Insert the pH probe, keeping the stir bar from hitting the probe.
- e) The pH of the solution should read less than 2.0<sup>3</sup> If the pH is less than 2.0, proceed with the titration step. If the pH is greater that 2.0 repeat the above steps using half the amount of sample used. A reagent blank, is required.
- f) After transferring the sample to a 100 ml titration beaker, titrate the sample using the 0.25 N NaOH to the end point using routine KA05 for 0.5 N HCl or KA04 for 0.1N HCl.

## (1) **REAGENTS & APPARATUS**

(a) Mettler DL70 ES Titrator with auto sampler tray, automatic stirrer and a 20 ml Burette with 0.01 ml precision in delivery.

<sup>&</sup>lt;sup>3</sup> See White, W.W. III; Cox, R. L.; Lapakko, K. A. STATIC-TEST METHODS MOST COMMONLY USED TO PREDICT ANCID MINE DRAINAGE: Practical Guidlinies for Use and interpretation p. 13

- (b) Calibrated Mettler auto titrator cups. (Cups are calibrated from 20 ml to 100 ml with 20 ml increments.)
- (c) Standard Sodium Hydroxide 0.25N spike solution Fisher Scientific Certified Standard.

## (2) PROCEDURE

- (a) Calibration:
  - (i) Titrant verification: (This is done when a new lot of Titrant is received. See section 4.)
- (b) Daily pH electrode Calibration:
  - (i) Obtain pH Buffer solutions pH 4, pH 7, pH 10.
  - (ii) Pour into the titrator cup approximately 60 mls.
  - (iii) Place sample cups in the auto sampler.
  - (iv) Select Analysis from Main Menu.
  - (v) Add routine (Press Run.)
  - (vi) Type KC01 (Press run twice)
  - (vii) Type in identity of buffers and press run after each buffer.
  - (viii) The titrator will then advance the sample tray and perform the calibration of the electrode.

- (c) Sample Preparation
- (d) Using the 100 ml calibrated Mettler Titrate Cups write the Lab # on the cup and pour prepared samples into the titration beakers.
- (e) Place samples in the autosampler tray. (Remember to count the number in the run including the QC samples.)
- (f) For QC samples: Write the Lab # on the cups and use the "chosen" QC sample to prepare and pour the original, duplicate, spike 1, spike 2, blank and blank spike. (A 50 mg addition of Calcium Carbonate is made to all spike samples prior to digestion.)
- (g) After the Daily Calibration is done running, return to the method screen.
- (h) Add routine (hit run)
- (i) Type KA05/KA04 and enter the number of samples in the batch and press run. The next screen is for typing in the sample id. After completing the sample ID table press run twice and the Titrator will start. (If you have more than twenty samples then you must empty the determined samples out of the autosampler and add the remaining samples. The maximum number of samples is 32.) The titrator will then proceede to sequentially determine each sample using the dynamic endpoint. (The titrator is restrained to have a pH of 7.)
- (j) Run reagent blanks by titrating the appropriate volumes of HCl with 0.25N NaOH.
- (3) Calculations:

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(a) Neutralization Potential (NP) in tons is calculated by the method using the equation below:

Tons Ca CO3/ Kton =  $\frac{\text{(Volume acid x Normality of acid - Volume base x Normality of base)} * 50}{\text{Sample Weight}}$ 

## IV. QUALITY CONTROL

- A. All sample runs will include a Known Control, a Duplicate, a Blank, and a Spiked Blank for at least every 20 samples.
- B. Duplicates must achieve an RPD of less than 35%, or the sample set must be reanalyzed.
- C. Known Control samples must be within statistical control limits as determined using the Shewhart control limits.
- D. Spiked Blanks must recover 80-120 % of true value

#### V. REFERENCES:

- A. EPA- 600/2-78-054. Field and Laboratory methods Applicable to Overburden and Minesoils. Industrial Environmental Research Laboratory, Office of Research and Development, U.S. EPA, Cincinnati.
- B. EPA-670/2-74-070. Mine Spoil Potentials for Soil and Water Quality. Nation Environmental Research Center, Office of Research and Development, U.S. EPA, Cincinnati.
- C. A.S.T.M. D744 Standard Method for Accelerated Weathering of Solid Materials Using Modisfied Humidity Cell.
- D. Society for Mining, Metallurgy, and Exploration, Inc. <u>Prediction of the Behavior of Mining and Processing Wastes in the Environment</u>. Richard W. Lawrence, 1990 Pgs 115-121.

- E. Operating manual for the LECO Sulfur Furnace.
- F. Operating manual for the Mettler DL70 Autotitrator.

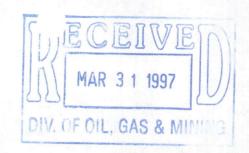
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## **AMMENDMENT**

(The reporting format is not typically placed in KEL SOPs. Therefore it will be listed as an ammendment to ABA SOP.)

The following analytes will be reported for Acid Base Accounting:

Total Sulfur
Hot water Extractable Sulfur
HCl Extractable Sulfur
HNO3 Extractable Sulfur
Residual Sulfur
Acid Potential
Neutralization Potential
Acid Base Accounting Calculation



## STANDARD OPERATING PROCEDURE #3

Sample Collection, Preservation, Chain of Custody, Archiving, and Quality Assurance

Ground Water Discharge Permit No. UGW350011

Appendix A

Revision 2 - January 1997

Prepared by: Kennecott Utah Copper

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#### STANDARD OPERATING PROCEDURE #3

Sample Collection, Preservation, Chain of Custody, Archiving, and Quality Assurance
Ground Water Discharge Permit No. UGW350011, Appendix A

#### 1.0 INTRODUCTION

The purpose of this document is to define standard operating procedures for the collection and analyses of tailings and other process samples collected to meet the requirements of Appendix A and B to the Ground Water Discharge Permit.

#### 2.0 SAMPLING

The procedures outlined in this section will be used for the documentation, collection, and storage of solid samples collected to satisfy the requirement of Appendix A and B.

Procedures referenced in the document "Standard Operating Procedures for Water Sampling" (SOP-WS), are attached.

## 2.1 Sample Collection

- 2.1.1 Collection of Samples From the Existing Impoundment
- Sample Location: Samples will be collected from locations as agreed upon by DWQ, on the existing impoundment. The location of each sample point will be marked, using a 3 foot lath, after the collection of the sample. The sample identification will be written on the lath with indelible ink. Sample locations will also be identified on a reference map of the impoundment.
- <u>Sample Collection</u>: Approximately 4 pounds of tailings will be collected at each site. A trench for the collection of these samples will be dug using a shovel. The sample will be collected using a stainless steel or plastic trowel or hand shovel. The sample will be identified, labeled, and documented as specified in section 2.2.
- <u>Sample Decontamination</u>: All sampling equipment contacting the sample will be decontaminated prior to sampling. Decontamination will consist of rinsing with detergent solution followed by a final rinse with deionized water
- A. Sampling Tailings Impoundment Embankment

Samples will be located so as to avoid previously sampled locations. Samples will be collected from 0 to 12 inches of depth below the surface. Plant roots and other debris will be removed from the sample.

### B. Samples from the Interior of Tailings Impoundment

Samples will be collected from the interior of the impoundment as the tailings impoundment is closed. These locations will be selected on a map prior to going to the Tailings Impoundment to avoid the introduction of a bias in the selection of the sampling site based on field conditions. These samples will be collected over the interval of 0 to 12 inches below the surface.

### C. Other Tailings Impoundment Samples

Other samples will be collected from the impoundment as samples of opportunity to investigate specific observed conditions. The locations and depths of these samples will be selected based on site conditions. The locations and conditions in the areas where these samples are located will be detailed in the field notebook as discussed in section 2.2.

### 2.1.2 Repeat Sampling

Samples will be collected from a number of process streams. These samples will be collected periodically as grab samples from the same sampling point. The process steams to be sampled are soilwater slurries. After sampling, excess water contained in the sample will be carefully decanted or filtered from the sample.

#### A. Tailings

Tailings samples will be collected from the Magna and Copperton concentrators and from the tailings cyclones. Samples of the Copperton tailings will be collected from Drop Box #6 as the tailings are discharged from the Copperton Concentrator. Samples of the Magna tailings will be collected from the launder crossing Highway 201. The locations of the samples to be collected from the cyclone stations will be identified when these structures have been constructed.

Samples at the Copperton and Magna concentrators will be collected by lowering a bucket into the drop box and collecting the slurry in the bucket.

#### B. Power Plant

Fly Ash from the Power Plant will be collected from the fly ash launder as it leaves the Power Plant. The sample will be collected in a bucket and the excess water decanted.

## C. Slag Tailings

The slag tailings will be collected from the tailings launder just prior to the slag tailings pump station. The sample will be collected in a bucket and the excess water decanted.

## D. Hydromet Plant

Samples of the Hydromet Plant slurry will be obtained using an in-line sample splitter. The sample will be a weekly composite sample obtained for operational purposes. A split of this slurry will be obtained for TCLP testing. Another portion of the sample will be allowed to settle and the excess water carefully decanted or filtered to obtain the solids fraction for use in ABA testing. Chain of custody procedures for this sample will not start until the sample is delivered to the Environmental Laboratory.

## 2.2 <u>Sample Documentation</u>

Field data will be entered into a field log book. Procedures for the preparation and use of a field log book are specified in Procedure DC-01 of the SOP-WS. The relevant sampling data will be entered into the Kennecott Sample Information Database. A copy of a document describing the entry of this data is attached.

### 2.2.1 Sample Identification

## A. Samples of the Embankment of the Existing Impoundment

One time tailings samples will be collected and identified using a field name. These field names, collected from transects located around on the sides of the impoundment, will be identified with a sequential number prefixed with the letters AT- and followed with the letters U, M, or L to designate the upper, middle, or lower portion of the impoundment transect. Other samples collected from the impoundment will be a sequenced number that uses TL as a prefix.

### B. Repeat Samples

Repeat samples will be identified using the sample identification procedures specified in Procedure DC-02 of the SOP for water sampling with the modification that the depth modifier will be used to indicate the type of analyses performed. The letter T will be appended to the structured sample name for reporting the results of total metals analyses. Similarly, the letter S will be used in reporting the results of the SPLP analyses and, the letter A will be used to report the results of the ABA procedures. This convention is needed to allow storage of the sample results on the mainframe. Table 1 provides the sample names, location, source, and type of material to be sampled.

## C. Kinetic Test Samples

Samples submitted for Kinetic sampling will be re-identified using sequential structured sample numbers starting with K000 and incrementing by 1 with each new sample. The sample result will then be referenced back to the original sample name.

### D. Other Samples

Other samples collected from the impoundment will be a sequenced number that uses TL as a prefix. Procedures used in the development of these sample identifiers are specified in the document "Kennecott Sample Information Database". A copy of this document is attached.

#### 2.2.2 Sample Containers

Sample containers will be as specified in Procedure DC-03 of the SOP for Water Sampling, except that containers used need only to be new, they do not need to be cleaned in the laboratory or rinsed with distilled water.

### 2.2.3 Sample Labeling

Samples will be labeled as indicated in Procedure DC-04. The suffix used to identify the type of analyses preformed will not be identified on the sample name.

### 2.2.4 Chain-of-Custody

Location and possession of the samples will be documented from the time of sampling until the sample is disposed of. Procedures for documenting the custody of the sample are specified in Procedure DC-04 of the SOP for Water Sampling.

### 2.2.5 Sample Preservation and Packaging

All samples will be air dried when received by the laboratory and stored in a dry environment. The maximum holding time for the sample is considered to be two years. All samples submitted for ABA analyses will be archived for a minimum period of two years after collection.

#### 3.0 ANALYSES

All samples will be air dried and homogenized by the laboratory prior to the start of any analytical procedures. All sample analyses will be done on the as received sample except for ABA analyses which will be run on an oven dried portion of the sample, ground to 200 mesh, and kinetic testing which will utilize a <-2mm particle size.

#### 3.1 Procedures

- A. Moisture content will be determined on the original sample prior to oven drying using the method described in "Methods of Soil Analyses," American Society of Agronomy, sixth printing, 1983; Part I, 7-2.2 pp.92-96. The moisture content will be reported with the results of the ABA testing.
- B. Samples submitted for Total Metals Analyses will be prepared using method 3060 and the analyses performed using EPA SW846 Method 6010 or 6020 for the following analytes: arsenic, cadmium, chromium, copper, lead, selenium, and zinc.
- C. SPLP analyses will be performed using EPA SW846 Method 1312 for the following analytes: arsenic, cadmium, chromium, copper, lead, selenium, and zinc.
- D. Analyses performed using ABA procedures will follow the "Modified Sobek Method" (SOP #1, #2, #3, and 4). Additionally, all samples submitted for ABA analyses should be analyzed for soil paste, pH, and conductivity. These measurements will be made on a saturated extract using the method discussed in "Methods of Soil Analyses," American Society of Agronomy, sixth printing, 1983; Part 2, 10-2.3.1 p.169. Soil paste and conductivity will be performed using methods 10-3.2.1 and 10-3.3, respectively.
- E. Kinetic testing will be performed using the "Kinetic Testing by the Humidity Cell Procedure" (SOP #5). As indicated in the procedure, the length of time required to complete the test could

range from 2 weeks to 1 year. It is anticipated that the duration of the test will typically be 20 weeks.

## 4.0 QUALITY ASSURANCE PROCEDURES

#### 4.1 Field

The field quality assurance program will include duplicates, reference, and blank samples. These samples will submitted to the laboratory at the rate of one sample in twenty.

The reference sample will be drawn from a large sample of underflow tailings material that has been thoroughly mixed and split into individual samples. This sample has been used in previous ABA investigations.

#### 4.2 Laboratory

#### 4.2.1 Total Metals and SPLP

Laboratory quality assurance programs for total metals and SPLP analyses will include, in addition to the QA/QC items specified by the method, a reference tailings sample. The replicate sample will be a specially prepared tailings sample that has been homogenized for use as a control sample by the laboratory. A split of this sample will be included in each batch of samples analyzed at the rate of at least 1 in every 20 samples. Sample runs in which the replicate sample does not fall within the 95% confidence interval of the mean of previous analyses will also be re-run. A quality assurance report will be provided for each sample run, which provides the results of duplicate and replicate sample analyses.

#### 4.2.2 ABA Procedure

The laboratory QA/QC program for the ABA Procedure will include:

- Duplicates (1 in 20 samples)
- Replicate (1 in 20 samples)

The replicate sample will be the same tailings material as used for the total metals replicate. A replicate sample will be included in each batch of samples analyzed at the rate of at least 1 for every 20 samples. Any sample run in which the duplicate samples do not achieve an RPD less than  $\pm$  35 percent will be re-run. Sample runs in which the replicate sample does not fall within the 95% confidence interval of the mean of previous analyses will also be re-run. A quality assurance report will be provided for each sample run which provides the results of duplicate and replicate sample analyses.

#### 4.2.3 Kinetic Testing

No duplicate and/or replicate testing is required. The required QA/QC is described in the methods.

### 4.2.4 Moisture Content, Soil Paste pH, and Soil Paste Conductivity

The QA/QC for these samples will be the same as identified for the ABA procedure.

#### 4.3 Detection Limits

Detection Limits for each of the analytical components are specified in Table 2.

# 4.4 Reporting

A separate Certificate of Analyses will be required for each type of analyses performed (Total Metals, SPLP, ABA, and Kinetic).

The Certificate of Analyses for the ABA results should include:

Moisture Content
Soil Paste pH
Soil Paste Conductivity
Neutralization Potential as Percent CaCO<sub>3</sub>
Neutralization Potential as tons CaCO<sub>3</sub>/1000 tons soil
Total Sulfur
HCl Extractable Sulfur
HNO<sub>3</sub> Extractable Sulfur
Residual Sulfur
Acid Potential as tons CaCO<sub>3</sub>/1000 tons soil
Acid Base Potential as tons CaCO<sub>3</sub>/1000 tons soil

The Certificate of Analyses for the Kinetic test will provide the weekly results of the measurements of pH, conductivity, acidity, alkalinity, and sulfate. The start date for each measurement will also be provided.

The interpreted results of all tests conducted within a given year must be reported to the DWQ prior to April of the following year. This will require that all laboratory work (including a quality assurance report) is completed and reported by February 15.

TABLE 1

Repeat Sample Descriptions

Sample Id.	Location	Frequency	Sample Source	Material Type	Analyses Required
MCP1480	Power Plant	Semi-Annual	Sampling Crew	Fly Ash	Total Metals, SPLP, ABA, and MC
SMP1481	Smelter	Semi-Annual	Sampling Crew	Slag Tailings	Total Metals, SPLP, ABA, and MC
SMP1482	Hydromet	Semi-Annual	KEL*	Hydromet Tailings	ABA, and MC**
BCP1483	Copperton	Monthly	Operations	Tailings	Total Metals, SPLP, ABA, and MC
MCP1484	Magna	Monthly	Operations	Copper Tailings	Total Metals, SPLP, ABA, and MC
TLP1485	Tailings (East Cyclone)	Monthly	Operations	Cycloned Tailings Underflow	Total Metals, SPLP, ABA, and MC
TLP1486	Tailings (East Cyclone)	Quarterly	Operations	Cycloned Tailings Overflow	Total Metals, SPLP, ABA, and MC
TLP1487	Tailings (West Cyclone)	Monthly	Operations	Cycloned Tailings Underflow	Total Metals, SPLP, ABA, and MC
TLP1488	Tailings (West Cyclone)	Quarterly	Operations	Cycloned Tailings Overflow	Total Metals, SPLP, ABA, and MC

## NOTES:

- \* KEL will obtain this sample from one of the operational monitoring samples collected from the Hydromet Plant
- \*\* Analyses for total metals and TCLP required for the permit will be obtained from the analyses obtain during routine operational monitoring of these materials.

### Abbreviations:

ABA = Acid base Accounting Procedures, MC = Moisture Content, SPLP=Synthetic Precipitation Leach Procedure

TABLE 2

Detection Limits

	Analytical Method					
Analyte	Total Metals	SPLP	ABA	Kinetic		
Conductivity (umhos)	NA	NA		10 (mg/l)		
Alkalinity (mg/l)	NA	NA	NA	10 (mg/l)		
Acidity (mg/l)	NA	NA	NA	10 (mg/l)		
Sulfate (mg/l)	NA	NA	NA	5 (mg/l)		
As	0.5 (mg/kg)	0.005 (mg/l)	NA	NA		
Cd (mg/l)	0.2 (mg/kg)	0.002 (mg/l)	NA	NA		
Cr (mg/l)	1.0 (mg/kg)	0.01 (mg/l)	NA	NA		
Cu (mg/l)	1.0 (mg/kg)	0.02 (mg/l)	NA	NA		
Pb (mg/l)	0.5 (mg/kg)	0.005 (mg/l)	NA	NA		
Se	0.5 (mg/kg)	0.003 (mg/l)	NA	NA		
Zn	1.0 (mg/kg)	0.01 (mg/l)	NA	NA		
Total Sulfur	NA	NA	0.01%	NA		
HCl Sulfur	NA	NA	0.01%	NA		
HNO3 Sulfur	NA	NA	0.01%	NA		
Residual Sulfur	NA	NA	0.01%	NA		
Neutralization Potential	NA	NA	0.01%	NA		
Acidification Potential	NA	NA	0.01%	NA		



# STANDARD OPERATING PROCEDURE #2

# Kinetic Testing by the Humidity Cell Procedure

Ground Water Discharge Permit No. UGW350011

Appendix A

Revision 1 - January 1997

Prepared by: Kennecott Utah Copper

Kennecott Environmental and Industrial Hygiene Laboratory 9600 W. 2100 S., Magna, UT 84044 SOP NO. KEL-5011.01 REVISION NO. 1 DATE 01-27-97 PAGE 1 OF 9

### STANDARD OPERATING PROCEDURE

# KINETIC TESTING BY THE HUMIDITY CELL PROCEDURE

APPROVAL:

Januar	130/97
Quality Assurance Manager	Date
Laboratory Director	Date

### I. TITLE

# KINETIC TESTING BY THE HUMIDITY CELL PROCEDURE

### II. SUMMARY

Kinetic tests such as the humidity cell have been developed to create conditions which favor oxidation so that acid production, if any, can be directly observed. Static testing (like ABA testing), determines the net potential for a sample to form acid, whereas the humidity cell measures the rate of acid formation. The balance of acid-forming and acid neutralizing reaction is reflected in the leachate chemistry. Tailings samples will be used. Tailings have a particle size less than 2 mm, so grinding is not necessary. The sample is placed in the bottom of the plastic humidity cell. Humid and dry air, and rinse water are passed over the top of the sample. The leachate is analyzed for pH, EC, Sulfate, Acidity, and Alkalinity.

#### III. PROCEDURES

### A. Kinetic Cell testing:

- 1. Reagents and Apparatus:
  - a. Plastic humidity cell. (PVC 8" Diameter x 4" height with an 8 " pvc perforated disk.)
  - b. Screen material teflon
  - c. Balance readability of 0.1 mg.
  - d. Weight Boats
  - e. Compressed Air and flow meters.
  - f. Thiobacillus ferrooxidans. (Kennecott will use 10 ml of KUC leach water.)

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### 2. Procedure:

- a. Weigh 833g of an air dried sample with a particle size of less than or equal to 2 mm.
- b. Rinse the sample into the kinetic cell chamber to a depth of 2 cm.
- c. The sample is inoculated with Thiobacillus ferroxidans.
- d. One day after the inoculation the sample is rinsed and the initial sample is analyzed.
- e. Dry air is forced through the humidity cell at a rate of 1 L/min for the first three days. Dry air is obtained by using a desiccant drying tube on the compressed air line.
- f. After three days of dry air, three days of moist air is added through the humidity cell. Moist air is created by forcing air through a porous stone immersed in a heated water bath.
- g. Each week, during the test the samples are rinsed with three pore volumes of deionized water. The water is added to the cell by using a peristaltic pump which delivers water at a rate of 2 ml/min.
- h. Pore Volume = (.40) \* volume of sample in the humidity cell.
- i. The humidity cell is allow to set for 1 hour before leachate is drained
- j. The leachate from the three pore volumes is analyzed for pH and EC readings immediately after collecting the effluent.
- B. pH and Electrical Conductivity(Kennecott Environmental Laboratory SOP 3002.00 & 3005.00):

- 1. Reagents and Apparatus:
  - a. pH meter.
  - b. Conductivity meter.
  - c. pH buffer reference solutions pH 4,7,10
  - d. Conductivity reference solution.

### 2. Procedure:

- a. Follow the manufacturer's recommendation for instrument calibration of the pH meter and conductivity meter.
- b. A portion of the leachate solution is placed in a beaker and is stirred by a magnetic stirrer. The combination pH and conductivity electrodes are immersed in the sample for a sufficient duration to obtain a stable reading.

# C. Alkalinity (EPA Method 310.1/Kennecott Environmental Laboratory SOP 3023.00)

- 1. Reagents and Apparatus:
  - a. Mettler Autotitrator.
  - b. Disposable beakers.
  - c. 0.5 N H2SO4 or HCl

### 2. Procedure:

a. A sample aliquot of 50 ml is poured into the disposable beaker.

- b. The beaker is placed on the autotitrator. The autotitrator is programmed according to the manufacturer's specifications for method 310.1, as Method ACD1.
- c. The sample is titrated to an pH end point of 4.5. The computer calculates the alkalinity as mg/L of CaCO3 based on the acid strength.

# D. Acidity (EPA Method 305.1/Kennecott Envonmental Laboratory SOP 3030.00)

- 1. Reagents & Apparatus:
  - a. Mettler Autotitrator.
  - b. Disposable beakers.
  - c. 0.25 N NaOH.

### 2. Procedure:

- a. A sample aliquot of 50 ml is poured into the disposable beaker.
- b. The beaker is placed on the autotitrator. The Titrator is programmed according to the manufacturers specifications for method 305.1.
- c. The sample is titrated to an pH end point of 8.2. The computer calculates the acidity as mg/L of CaCO3 determined by the base strength.

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# E. <u>Sulfate (EPA Method 375.2/ Kennecott Environmental Laboratory SOP 3013.01)</u>

• EPA Method 300 or EPA 375.1 maybe substituted for EPA 375.2

### 1. Reagents and Apparatus:

- a. Automated analytical equipment, RFA II, with 460 nm filter and sulfate manifold according to manufacture's specifications.
- b. Ion exchange column, 10 cm, packed with 35-50 mesh Na form resin.
- c. Barium Chloride solution, 1.526 g BaCl<sub>2</sub>-2H<sub>2</sub>O in 1 liter DI water.
- d. Hydrochloric Acid, 1.0 N.
- e. Sodium Hydroxide solution, 0.18 N.
- f. Brij-35, 30% solution.
- g. Methylthymol blue (MTB) reagent, 0.0676 g MTB, 10 ml BaCl<sub>2</sub> solution, 28 ml DI water, 1 ml 1.0 N HCL, 1 ml Brij, q.s. 200 ml reagent alcohol (ethanol).
- h. Buffered EDTA cleaning solution.

### 2. Procedure

- a. Set up the manifold by following the general procedures prescribed by the manufacturer.
- b. Using the Labtronics software for RFA analysis, prepare standard curves by plotting peak heights of calibrated standards. Procedures for this step can be found in the manufacture's manuals.

- c. Compute sample concentration by comparing sample peak height with standard curve.
- d. After use rinse MTB and NaOH reagent lines with DI water for a few minutes, then with EDTA solution for 3 minutes, then again with DI water for 10 minutes.

#### 3. Standards

a. From a 1000 mg/l stock certified sulfate standard, perform dilution by carefully pipetting 1, 2, 4, 6, 8, and 10 ml into a final volume of 100 ml using class A volumetric flasks or by another means.

## 4. Quality Control

a. Independent source from calibration standard prep. QC1:
Analytical weigh 35.5 g CaSO<sub>4</sub> into a 500 ml volumetric flask,
dilute to volume with Type I water. QC2: Volumetrically pipette
0.5 ml 1000 mg/l SO<sub>4</sub> standard into a 100 ml volumetric flask,
dilute to volume with Type I water. Spike Standard: Fortify
individual 2.0 ml sample cups with 0.040 ml 1000 mg/l SO<sub>4</sub>
standard, make certain that spiked samples are well mixed.

### IV. TERMINATION OF TESTING

The length of time for kinetic testing of individual samples will be based on the following criteria:

- A. If the pH of the humidity cell leachate remains at or below 3.5 for 2 weeks the test can, at the discretion of Kennecott, be terminated.
- B. All other tests will be conducted for 20 weeks unless the following conditions are met:
  - 1. If the pH of the humidity cell leachate drops by 2 or more units between weeks 11 and 20, the test will be extended until the pH stabilizes. pH will be considered stable if it does not vary by more than 0.5 units

during the most recent 4 weeks of testing or drops below 3.5. If the pH drops below 3.5 for 2 weeks, the test can be terminated at the discretion of Kennecott.

2. If the average rate of release of sulfate in leachate from the humidity cell between weeks 16 and 20 is greater than 50 mg/Kg of an air dried solid sample, and the corresponding calculated rate of acid released by oxidation of pyrite would be enough to deplete the neutralization potential within 52 weeks, the test shall be extended until acidic conditions develop, or until 52 weeks have passed since initiation of the kinetic testing.

## V. Reporting

A. Standard reports will be reported in dry weight basis. (The Dry weight will be obtained using the % Moisture in the ABA analysis.) The report will include a detailed sample description, the static test results for the sample, and weekly humidity cell results. Weekly analyses of solutions include pH (standard units), electrical conductivity(micromhos/cm), sulfate mg/L, acidity (mg/L as CaCO3), alkalinity (mg/L as CaCO3), and net alkalinity (alkalinity minus acidity in units of mg/L as CaCO3).

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